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# Physicochemical and crystalline properties of standard maize starch hydrothermally treated by direct steaming

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ABSTRACT

The changes in physicochemical properties of standard maize starch (SMS) by three hydrothermal treatments; DV-HMT (Direct Vapor-Heat Moisture Treatment), RP-HMT (Reduced Pressurized-Heat Moisture Treatment) and DIC (instantaneous controlled pressure drop) were investigated at different processing conditions; steam pressure (SP) varied from 1 to 3 bar during 20 min. Starch was steamed by direct contact, whose interest was to intensify the heat transfer phenomenon but also the water transfer. The physicochemical changes of SMS depended on process conditions and their extent followed this order: DIC > RP-HMT > DV-HMT. All treatments significantly increased gelatinization temperatures and decreased the enthalpies, confirmed by loss of granules birefringence. From 2 bar, the crystalline structure changed from A-type to $V_h$-type, revealing formation of amylose-lipid complexes during steaming. The results clearly showed that the particle size distribution depends on the melting extent of crystalline structure during treatment. At severe processing conditions the melted fraction increased and more complex aggregates of different sizes have been formed.

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## 1. Introduction

Starch which is a renewable biopolymer and one of the most abundant carbohydrates reserve, constitutes a fundamental material for food and non-food use due to its large physicochemical properties. Generally, modification of starch is carried out to enhance the positive characteristics and eliminate the shortcomings of native starches. Heat-Moisture Treatment (HMT) modifies the physicochemical properties of starch without destroying the granular structure. Starch is heated at high temperature ( < 140 °C) but with restricted moisture between 13 and 30 g/100 g (Arns et al., 2015;Kong et al., 2014; Sui et al., 2015 Zen, Ma, Kong, Gao, & Yu, 2015), above its glass transition temperature and below the gelatinization temperature for times of few minutes (Zarguili, Maache-Rezzoug, Loisel, & Doublier, 2006; Lim, Chang, & Chung, 2001) to hours (Ji et al., 2015; Kong et al., 2014 Wang, Zhang, Chen, & Li, 2016). The comprehension of starch phase transitions is extremely important in the food processing operations. Starch with a semi-crystalline structure has two phase transitions which are likely to occur during HMT treatment: the glass transition that concerns the amorphous phase (mainly the branching regions of the amylopectin and most of amylose chains) and melting of crystallites (formed by adjacent short chains of amylopectin intertwined into double helices). The melting temperatures of crystallites depend strongly on the moisture content. For moisture content higher than 60% (w/w), only one single endotherm occurs over a constant temperature range, reflecting the loss of semi-crystalline order and transition temperature is referred to as gelatinization temperature. For lower moisture contents, multiple transitions occur that reflect melting and recrystallisation processes appearing simultaneously during heating (Biliaderis, Page, Maurice, & Juliano, 1986;Maache-Rezzoug, Zarguili, Loisel, Queveau, & Buleíon, 2008). The transition temperatures, designated as melting temperatures, increase and multiple endotherms are formed to the detriment of gelatinization endotherm. The melting term is then preferred to describe the thermal transitions during heating at low or intermediate moisture contents. In a previous work (Maache-Rezzoug et al., 2008), we have shown that the low moisture content (i.e. less than 35%) prevailing during DIC treatment were not sufficient to ensure that the gelatinisation happens. Many studies have been carried out

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on physicochemical properties of starch granules after HMT treatment. The properties of treated starches depend on the starch origin (Maache-Rezzoug, Zarguili, Loisel, & Doublier, 2010; Collado, & Corke, 1999; Gunaratne, & Hoover, 2002; Hoover and Manuel, 1996; Hoover and Vasanthan, 1994; Kulp, & Lorenz, 1981) and treatment conditions used (Kong et al., 2014; Sui et al., 2015; Takaya and Nishinari, 2000).

Some investigations (Chung, Liu, & Hoover, 2009; Gunaratne, & Hoover, 2002; Hoover & Manuel, 1996 Malumba, Massaux, Deroanne, Masimango, & Bera, 2009;Vermeylen, Goderis, Reynaers, & Delcour, 2006) have shown that the characteristic temperatures and enthalpy of gelatinization (ΔH) of treated starches by HMT were mainly influenced by applied moisture level and temperature. Maache-Rezzoug, Zarguili, Loisel, Doublier, & Buléon (2011) observed similar effects after DIC treatment, except that the gelatinization temperature range was narrowed as observed with annealing (Hublin, 1994; Jayakody & Hoover, 2008). The authors suppose that the DIC treatment has led first the melting of crystallites of low cohesion, those requiring less energy and the remaining crystallites in the residual structure have greater cohesion. Bahrani, Loisel, Doublier, Rezzoug, & Maache-Rezzoug (2012) have already shown during the steaming of SMS by three HMT processes that the heating of starch granules is the result of transfer of latent heat of steam condensation by direct contact with saturated steam that also contributed to water transfer. Starch temperature rises from room temperature to steam equilibrium temperature. The higher the difference in temperature, the higher the quantity of condensed water, which causes increasing of starch moisture content (Zarguili et al., 2006). Direct Vapour-Heat Moisture Treatment, termed as DV-HMT treatment by our team, consists in heating up starch by saturated steam injected from atmospheric pressure up to processing steam pressure level. This treatment belongs to HMT according to the definition of Sair and Fetzer (1944). As regards to RP-HMT (Maruta et al., 1994) and DIC, the two processes begin by the setting up of vacuum prior to injection of saturated steam, contributing to intensify its diffusion into the product by reducing the air resistance. Consequently, the time required to reach steam equilibrium temperature is shortcut (Bahrani, Monteau, Rezzoug, Loisel, & Maache-Rezzoug, 2014). Only DIC process contains a final step of abrupt decompression which carries out towards the vacuum (Rezzoug, Maache-Rezzoug, Mazoyer, & Allaf, 2000) instead of atmospheric pressure as for DV-HMT and RP-HMT.When pressure suddenly decreases, the autovaporization which is an adiabatic transition occurs and water rashly escapes accompanied by a rapid cooling, whose value stabilizes at the equilibrium temperature of final pressure. The mechanical action and the intense shear of granules contribute to alter the crystalline structure; some mechanical energy is converted into internal energy (Huang, Lu, Li, & Tong, 2007). Bahrani et al. (2014) and Zarguili, Maache-Rezzoug, Loisel and Doublier (2009) showed during DV-HMT and RP-HMT/DIC processes that the treatments were homogeneous; the absence of moisture gradients between the different starch layers was evidenced during monitoring of spatial evolution of moisture content in the thickness direction at different processing times. The presence of initial vacuum in the case of RP-HMT/DIC, contributes to generate a more significant amount of condensed steam than for DV-HMT, the moisture content were 24 and 20%, respectively. For a same SP, the volume occupied by air in the reactor is replaced by an equivalent volume of steam. The objective of this study was to investigate the effect of three hydrothermal processes; DV-HMT RP-HMT and DIC on the physicochemical changes of SMS. The impact of processes, mainly the additional vacuum steps before and after steaming phase of starch granules, on the thermal transitions, structural characteristics and morphological properties were investigated at different processing temperatures.

## 2. Materials and methods

### 2.1. Raw material

Standard maize starch (SMS) at residual moisture content of 14% (g $H_2O$/g db) and with relative weight percentages of amylose and amylopectin of 26 and 74%, respectively, was supplied by Roquette Frères (Lestrem, France).

### 2.2. Moisture content measurement

Moisture content was determined by air oven at 105 °C during 3 h, according to the A.O.A.C (1999) standard method. As the evaporation phenomenon during the pressure release can disturbs the real estimation of absorbed water, the brutal decompression of DIC was replaced by a gradual depressurization to atmospheric pressure and thus the moisture content in RP-HMT and DIC are similar. Three replicates were performed and the standard deviation was about 0.2% (dry basis).

### 2.3. Experimental set-up

The detailed procedure and equipment have been described previously by Bahrani, Loisel, Maache-Rezzoug, Della Valle, & Rezzoug (2013). Briefly, the experimental setup is composed from: a processing reactor, where the sample was treated at high SP/temperature, a vacuum system which comprises mainly a stainless steel vacuum tank with a volume of 1600 L, 130 fold greater than that of the reactor (12 L), a vacuum pump, and a steam generator supplying steam into the reactor.

### 2.4. Hydrothermal processes

The hydrothermal treatments were performed in pilot scale at fixed SP for 20 min. SMS (5 mm thickness) was placed in an aluminium rectangular container in reactor at a residual moisture content without any hydration step (Bahrani, 2012). Five SP levels were investigated: 1, 1.5, 2, 2.5 and 3 bar, corresponding to saturated steam temperatures of 100, 111, 120, 127 and 133 °C, respectively.

In DV-HMT, saturated steam was injected from atmospheric pressure up to processing SP level and maintained during 20 min. Processing time begins when SP reaches setting pressure. This step is followed by an abrupt decompression towards atmospheric pressure. While for RP-HMT and DIC reduced pressure of 50 mbar was established in the reactor before injection of live steam at fixed SP. After the maintaining phase, the abrupt decompression is performed towards atmospheric pressure for RP-HMT and vacuum pressure (50 mbar) for DIC. The presence of initial vacuum for both RP-HMT and DIC processes contributes to accelerate the transfer phenomenon associated with simultaneous heat and mass transfer by comparison with DV-HMT. At the beginning of hydrothermal treatment, the distribution of temperature field is non-homogenous, and it becomes homogenous throughout the material when equilibrium temperature is reached.

### 2.5. X-ray diffraction

Native and treated starch were equilibrated at 95% relative humidity (RH) using a saturated barium chloride solution, and the X-ray diffraction pattern was measured with Inel (France) X-ray equipment at 40 kV and 30 mA. The diagrams of diffraction were recorded according to the method of Debye-Sherrer in transmission. Cu $K_{\alpha 1}$ radiation ($\lambda$=0.154 nm) was selected using a quartz monochromator. A curved position-sensitive detector CPS 120 was used to monitor diffracted intensities in the 0–120° 2θ range.

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